LLNL-TR-661706

LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

Summary of DKDP boule fracture tests

T. Suratwala, C. Thorsness, T. Land, M. Evans, K. Fogle, J. Kimmons, K. Kishiyama, R. Steele

September 30, 2014

Disclaimer

This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

Interdepartmental letterhead
Mail Station L-491
Ext: 2-1884

NATIONAL IGNITION FACILITY
PROGRAMS DIRECTORATE

NIF0091695/adf

MEMORANDUM – November 15, 2002

TO: File

FROM: T. Suratwala, C. Thorsness, T. Land, M. Evans, K. Fogle, J. Kimmons, K. Kishiyama, and R. Steele

SUBJECT: Summary of DKDP boule fracture tests

Introduction/ Summary

One of the technical challenges for the success of DKDP rapid crystal growth is to obtain a large crystal boule without it thermally fracturing at the end of a growth run. A typical high-temperature DKDP crystal growth run ends at a temperature of ~50-55^{o}C. At this time the crystal is still in solution. The solution then needs to be removed, and the temperature of the crystal needs to be lowered to RT without crystal fracture. In our previous analysis (based on small size thermal shock experiments), we had established that in order to prevent fracture in DKDP crystals, the average temperature in the crystal minus the surface crystal temperature at the 2D surface edge (ΔT) should be $\leq 1^oC$ [1]. Also, we calculated that at the end of a crystal growth run, the largest temperature drop occurs during the solution removal due to evaporation off the crystal surface [1].

In this study, a series of large-size DKDP boule thermal fracture tests were performed to validate the small-scale tests and to simulate the cooling of a crystal at the end of a DKDP rapid growth crystal run. The results of these tests suggest that a $\Delta T \leq 2.9\text{-}3.9^oC$ will not lead to fracture: hence the crystal boules are more resistant to fracture than our previously determined criteria. The reason for the difference may be due to the differences in flaw size between the small size samples (saw cut surfaces) vs the large boules (partially dissolved -blunted) surfaces. Regardless, to be conservative, we recommend that the original criteria of $\Delta T \leq 1^oC$ be maintained at the outer 2D edge of the crystal at all times*. This criteria will provide an upper bound to the cooling rate of the crystal which is dependent on the final crystal size. Also, to meet this criteria, the following conditions are recommended to minimize the chance of thermal fracture at the end of a crystal growth run: 1) input water-saturated, temperature-matched air into the growth tank as the salt solution is removed; 2) heat and insulate the lid of the growth tank; and 3) heat the Al shaft outside the growth tank. The use of the following conditions has been successful in cooling a Pilot Production DKDP Crystal FD-18 without fracture.

[1]Note that an additional ΔT constraint has also been implemented due to the chance of failure at the weaker regeneration interface of the crystal boule (see NIF0090381; November 6, 2002).

Experimental/ Results /Discussion

A series of large DKDP crystal boule (~85-280 Kg) fracture tests were performed. In these tests, the crystal boules were placed in a DKDP rapid growth tank. The crystals were heated to 60^{o}C and cooled under various conditions and at various rates.

A temperature-controlled sparger (in which air is bubbled through bubbler in a temperature controlled water bath to saturate the air) was fabricated in order to provide the water-saturated, temperature-matched air into growth tank as the solution is removed [2,3]. A photograph of one of the spargers is shown in Fig. 1a. A series of thermocouples were placed in the growth tank to monitor the temperature of the crystal during cooling. The locations of the thermocouples are shown in Fig 1b, which shows a schematic of the setup.

A summary of the tests performed on the large size crystal boules are shown in Table 1. A detailed account of experimental for each of the individual tests can be found in the crystal growth notebook [4], and hence is only briefly described here. In the first four tests, ½ of crystal FD-16 (a horizontal grown crystal, 85 kg) was placed in the growth tank and heated to ~60^{o}C in air. The water bath was then programmed to cool at 15, 30, 100, and 200^{o}C/day. The measured temperatures at the various locations are plotted as a function time in each for each of these tests in Fig. 2. The crystal fractured after cooling at 200^{o}C/day (see Fig. 3).

The calculated 3D ΔT is also reported in Table 1. Two ΔT's were reported: in the first case, heat transport from the bottom crystal surface is the same as all the other crystal surfaces, and in the second case, heat transport from the bottom surface is proportional to the ratio of the areas of the bottom surface of the platform with that of the bottom surface of the crystal. We currently believe that the second case is more valid and will use those values for the remainder of this discussion.

Fig. 4 is plot of the calculated ΔT for all the DKDP thermal shock experiments performed to date. The results from the large boule fracture tests suggest that a $\Delta T \leq 2.9$-3.9^{o}C will not lead to fracture. The crystal boules appear to be more resistant to fracture than our previously determined criteria ($\Delta T \leq 1^o$C) based on small size DKDP fracture tests [1]. The reason for the difference may be the differences in flaw size between the small size samples (saw cut surfaces) vs the large boules (partially-dissolved (blunted) surfaces). Regardless, to be conservative, we recommend that the original criteria of $\Delta T \leq 1^o$C be maintained at the outer 2D edge of the crystal at all times.

In test #5, the 2nd ½ of FD-16 was placed in the crystal growth tank and heated to ~60^{o}C just as in the previous tests. Separately, water was heated in an auxiliary tank to ~60^{o}C and then input into growth tank to submerse the crystal and Al platform above the top horizontal ledge. The solution was then removed and the air from the sparger was input simultaneously. Also, during cooling, the Al shaft was heated with heating tape to minimize heat loss from the growth tank through the Al shaft. The crystal was cooled at a modest 15^{o}C/day and did not fracture. However, the crystal did dry during cooling suggesting that evaporation off the crystal was still taking place. The goal of inputting saturated air into the growth tank was to prevent/minimize evaporation off the crystal surface to cause significant heat loss. The evaporation off the crystal surface can be explained by the cool lid temperature. Hence, solution evaporated off the crystal surface and then condensed on the lid, even though the environment had 100% relative humidity.

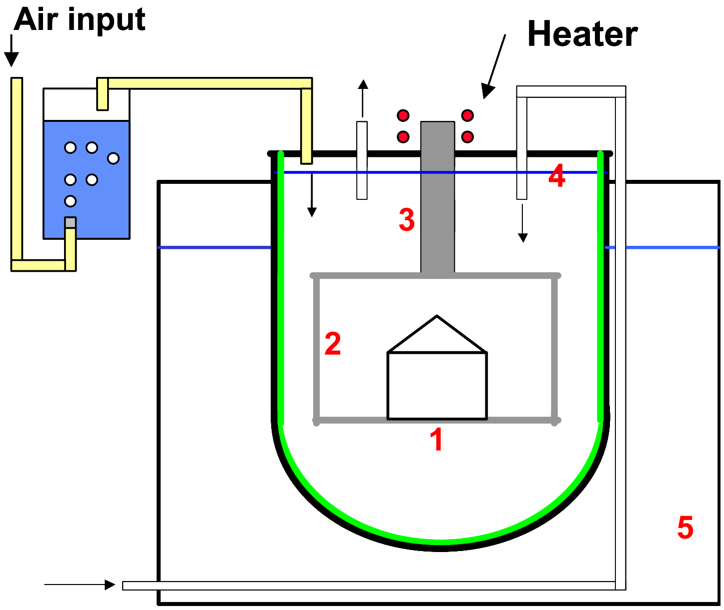
In test #6, we repeated test #5 but now placed water-heated Cu lines on the lid of the tank to ensure that the lid was not cooler than the crystal surface during cooling. As a result, the evaporation off the crystal surface was greatly reduced.

Test #7 was performed on at the end of a pilot production run (FD-18 (~280 kg)). At the end of the growth run, the following was performed: 1) solution was removed while the sparger input water-saturated, temperature-matched air, 2) the lid of the tank was headed using water Cu lines, 3) the Al shaft was heated, and 4) the crystal was cooled at 3^{o}C/day. The crystal was successfully cooled to room temperature and removed from the growth tank without fracture. The above conditions are recommended in the cooling of crystal boules in future growth runs.

References

[1] T. Suratwala, C. Thorsness, R. Steele, “Thermal Fracture of DKDP crystals” NIF0082445 (November 6, 2002).
[2] Version 1 was put together by Rusty Steele and Kevin Fogle.
[3] Version 2 was put together by Mark Evans, Kevin Fogle, and Aaron Nottingham.
[4] T. Land, Crystal Growth Notebook for Tank E, Crystal Growth Lab (2002).

(a)



(b)

Figure 1. (a) Photograph of sparger; **(b)** schematic of setup used for the thermal shock experiments. The numbers represent the locations of the thermocouples: (1) bottom of crystal, (2) platform arm, (3) platform shaft, (4) head space, and (5) bath temperature.

Table 1: Summary of DKDP crystal boule thermal shock test.

Test #	Crystal	(Size) (cm x cm x cm) Mass	Heating rate (°C/day)	Equilibration Time (hrs)	Cooling rate (Program) (°C/day)	ΔT (°C)	ΔT* (°C)	Heat Al platform?	Result
1 (No H_2O)	DKDP ½ FD-16	(77x24x19) 85kg	50	17	15	0.8	1.0	No	NO FRACTURE
2 (No H_2O)	DKDP ½ FD-16	(77x24x19) 85kg	50	17	30	1.5	2.0	No	NO FRACTURE
3 (No H_2O)	DKDP ½ FD-16	(77x24x19) 85kg	50	17	100	2.9	3.9	No	NO FRACTURE
4 (No H_2O)	DKDP ½ FD-16	(77x24x19) 85kg	50	17	200	4.7	7.2	No	FRACTURED!
5 (solution + sparger)	DKDP 2nd ½ FD-16	(77x24x19) ~85 kg	50	17	15	0.7	1.0	Yes	evaporation due to lid cooling
6 (solution + sparger) +lid heating/insulation	DKDP 2nd ½ FD-16	(77x24x19) ~85 kg	50	17	15	0.7	1.0	Yes	Evaporation greatly reduced
7 (solution + sparger) +lid heating/insulation	DKDP FD-18	(55x55x55) ~280 kg	na	na	3	0.3	0.4	Yes	No Fracture

T. Suratwala

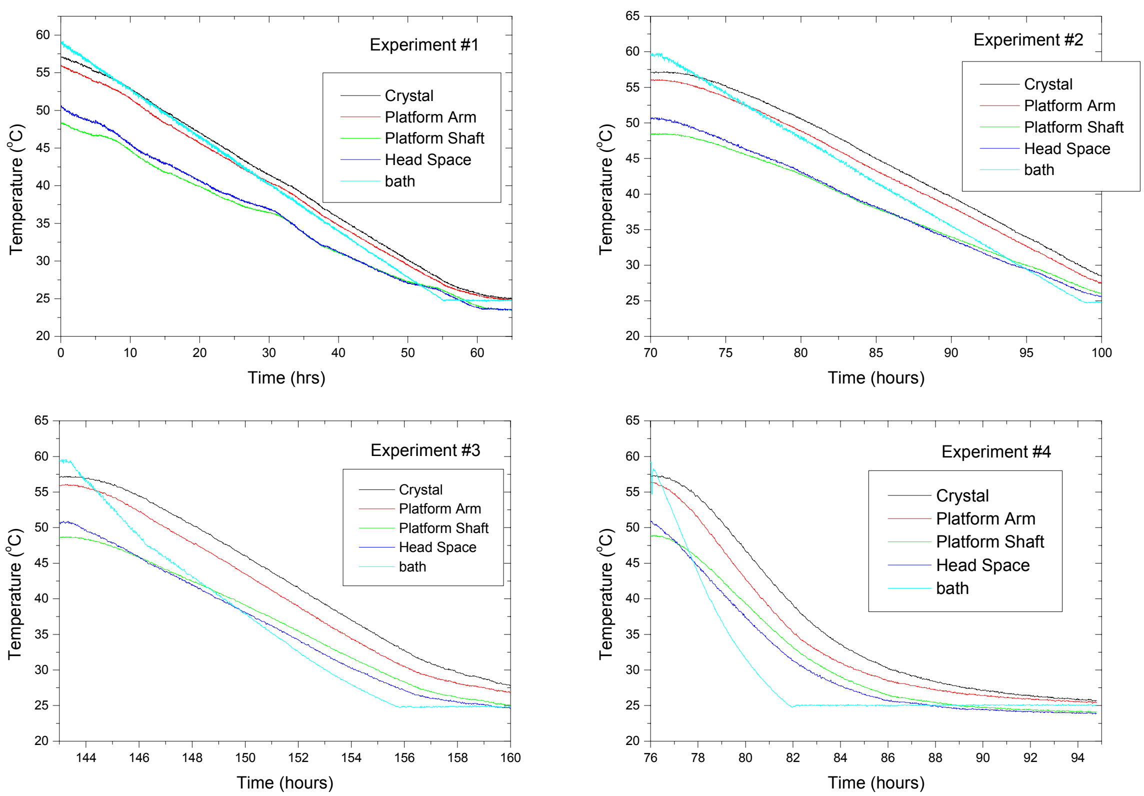


Figure 2: Measured temperature during the cooling of ½ of FD-16 in the first four thermal shock test in air. Crystal fracture occurred in test #4.

Figure 3. Photograph of ½ FD-16 after cooling the crystal at a programmed rate of 200°C/day. ΔT=7.3°C.

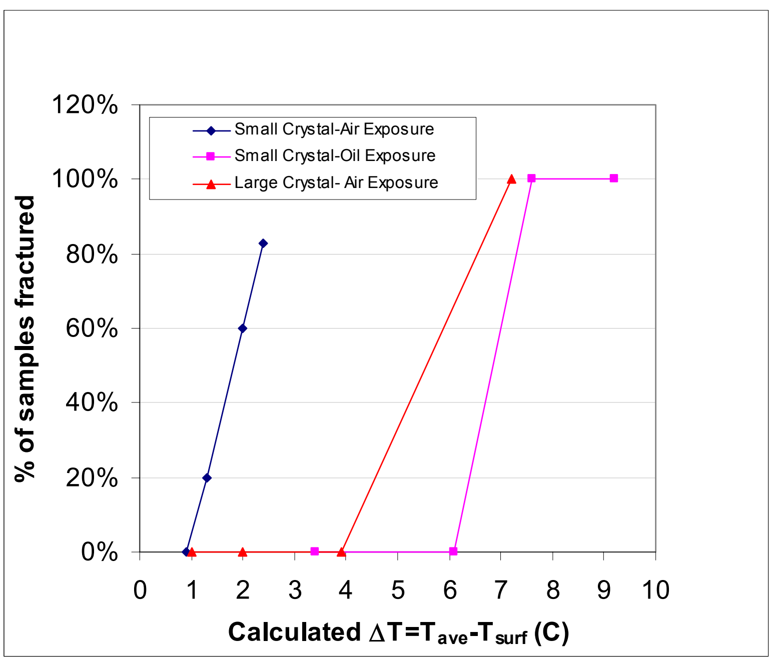


Figure 4. Comparison of all DKDP thermal shock experiments.